

Tantalum and Niobium – the keys to the markets of the future

Tantalum is among the rarer elements in the earth's crust. It ranks between thorium and caesium in frequency and is therefore rarer than lanthanum, lead, gallium or boron. Even niobium, which always occurs in association with tantalum, is ten times more available.

Tantalum was discovered in 1802, when A.G. Ekeberg separated the oxide of a new element from a Finnish mineral similar to columbite. The extremely stable oxide did not form a salt even when exposed to an excess of acid.

Ekeberg named the new mineral tantalum because he compared the behavior of the metal – which could not become “saturated” with acid – to Tantalus, the king of Phrygia in Greek mythology who was punished by Zeus for serving his murdered son, Pelops, at a feast for the gods to test their omniscience. Tantalus was con-demned to Hades where he had to stand eternally in water that receded when he bent down for a drink. The word tantalize is derived from his name.

Tantalus' daughter Niobe did not fare much better. She committed the unforgivable sin of boasting about her 12 children to Leto, Zeus' consort and mother of Apollo and Artemis. The gods were angered by this outrage and killed all of Niobe's offspring, whereupon she turned to stone while bewailing the loss of her children.

At nearly the same time as tantalum's discovery in 1801, Hatchett identified niobium in the form of its oxide. He named the element “columbium” after the place in North America where it was found. Niobium was later mistaken for tantalum, until Rose isolated the pure chloride and named the element on which it was based niobium. In 1864 Blomstrand succeeded in identifying the metal conclusively by reducing its chloride in a hydrogen atmosphere.

For many years, the name columbium was given the same status in scientific circles as niobium. Not until 1949 was the name niobium recommended by the International Union of Pure and Applied Chemistry, better known as the IUPAC, as the accepted international designation.

Niobium and tantalum usually occur in nature in association with iron and manganese. However, the metals are rarely found in concentrated amounts in the few and widely scattered places of discovery. Instead they are frequently found with other elements, such as tin or even rare earths.

The economically most important deposits are in Australia, Brazil, Canada, Thailand, Malaysia and China. In addition, there are exploited deposits in Western and Central African countries, CIS countries, Central and South America, Burma and Europe, although Germany (Kaiserstuhl), Norway and Finland are interesting from a purely mineralogical point of view.

Metals with interesting properties

Tantalum is a platinum gray, hard and very ductile metal that can be rolled and forged. In terms of tensile strength, elasticity and heat conductivity, tantalum is comparable to the average types of steel. Tantalum has a density of 16.6 g/cm³ and liquifies at a temperature of 3,000° C.

In more current literature, tantalum's boiling point is listed as 5,427° C, give or take 100° C as a factor of uncertainty, which is the case of all materials with extremely high boiling points. Older scientific reference books cite much higher boiling points of around 6,100° C .

Although a nonprecious metal, tantalum is quickly covered with a protective oxidic coating when exposed to air, which makes it extremely resistant to chemical corrosion. Thus the metal can only be corroded by hydrofluoric acid, hot fuming sulfuric acid, hot chlorine gas or fluorine, sulfur and liquefied alkalis.

Niobium is an air-resistant, light gray, forgeable metal, whose polished surfaces are shiny white. At 8.58 g/cm its density is far lower than that of tantalum. Depending on the source of reference, the metal's melting point varies from 2,468° to 2,497° C and the boiling point from 4,927° to 5,100° C.

Like tantalum, niobium resists most acids. Hydrofluoric acid, hot concentrated sulfuric acid and hot alkali lye corrode the metal. Niobium oxidizes when heated in air at temperatures above 300° C. Hydrogen and nitrogen are absorbed at temperatures over 250° to 300° C, forming very brittle, solid solutions. This is why processing niobium at high temperatures has to be performed in an inert gas atmosphere or in a vacuum.

The human body also contains niobium metal, although only in small quantities of 100 mg. Nothing is known about its physiological significance.

Love at second sight

Unlike molybdenum and tungsten, areas in which H.C. Starck is among the first generation of producers, the company's involvement with tantalum and niobium was more like "love at second sight." Nevertheless H.C. Starck led the pack of second generation producers, who initiated their activities in the United States beginning in 1955.

From preliminary tests to the first patent

H.C. Starck began its first tests to recover tantalum and niobium from residues left over from the concentration of hardmetal scrap during World War II. The hardmetal scrap came from the United States and, unlike German scrap, contained tantalum. Unfortunately, it is not possible to reconstruct the actual outcome of these preliminary experiments. These early activities were given new impetus in 1948, when, under Dr. Herrmann Lang's supervision, Horst Meyer started working on tantalum recovery from these residues. Because of his extremely dedicated work, he was nicknamed "tantalum Meyer" in the company.

After successful preliminary tests, a "task force" for tantalum was set up in 1949. At that time two products were of interest: tantalum oxide, which served as a starting material for tantalum carbide, and tantalum metal. The base material for both products was potassium fluorotantalate, K_2TaF_7 ..The tantalum/niobium separation was achieved in those days by the de Marignac process involving fractional crystallization – a method that makes use of the differences in solubility of the potassium double fluorides.

H.C. Starck, however, never worked with the de Marignac process. The double fluorides that were required for the production of oxide and metal were initially supplied entirely by the Germany company “GfE Gesellschaft für Elektrometallurgie,” which was among the first generation tantalum producers.

Another activity pursued by Elchem, the research institute jointly established by H.C. Starck and GfE in 1951, should be mentioned in this context: the development of an extraction process that provided the basis for the first, albeit very small, liquid-liquid extraction unit set up at the Goslar plant in 1955. The basic research for this project originated in the U.S. Bureau of Mines in Denver, Colorado. Important impulses also came from Murex in the U.K.

Production activities in those days were concentrated primarily on tantalum metal, which resulted in a patent being granted for tantalum metal production by reducing potassium fluorotantalate with sodium vapor. According to the patent, which was based on a discovery by Dr. Lang, the process was conducted in nickel pipes in which double fluoride and sodium metal were separately induced to react in boats. Some 100 to 200 grams of tantalum metal were produced in each batch.

Greater quantities were obtained by simply increasing the number of tubular furnaces. However, the H.C. Starck experts soon agreed that largescale production would not be possible with this method. Tantalum metal was supplied to Siemens, which by that time had shut down its chemical division, but still manufactured mill products.

Tantalum oxide production is introduced

Early attempts to produce tantalum oxide from double fluoride using wet chemical methods were less successful. The resulting product always contained large amounts of alkali and was unsuitable for further processing to carbide.

The separation of the bonded potassium and the production of pure tantalum oxide first succeeded after the base oxide obtained from the precipitation of double fluoride solution with soda was subjected to a sodium hydroxide fusion. This process was patented in 1951.

Rotating slanted cast iron retorts in which the fusion step took place were the basis of this process. Following decomposition of the oxide, the melt was poured into pans and after solidification, the product was dissolved in water. Impurities of iron were dissolved by acidifying, and after an additional washing process, high-purity tantalum oxide was separated off as filter residue. This led to a respectable output of some 10 tons of tantalum oxide per month by 1952. Similar to the production of ST grades of tungsten (see the chapter on tungsten), carbide production in circular furnaces took place in chamotte crucibles.

The concentration of tin slags

The year 1954 represents a particularly important milestone in the history of tantalum chemistry in Goslar. H.C. Starck was the first company in the world to introduce a process for the production of synthetic concentrates from tin slags. These slags were in fact waste products from tin smelting.

Besides niobium, these black, glass-like calcium aluminum silicate slags can contain up to 18 percent tantalum oxide. The niobium contents, however, can vary greatly, largely depending on the country of origin.

Before tantalum became lucrative as a metal, tin slags were used for many years as filling material in road construction and also to make embankments. In the course of tantalum's development, these old deposits of tantalum slags became at least temporarily interesting as "tantalum claims," depending on the metal price at the time.

Using H.C. Starck's proprietary process, a ferroalloy is produced from tin slags. This is done in combination with fluxes and by adding reducing agents. The concentrate is later produced from this alloy.

The melt containing tantalum and niobium was subsequently crushed and treated with hydrochloric acid to extract the iron impurities. A sodium hydroxide treatment was used to remove the silicic acid, and the remaining "raw" concentrate of tantalum and niobium carbide was roasted in so-called flat furnaces. Parallel to these activities in Goslar, progress was made in Laufenburg on the further development of a process for the production of synthetic concentrates.

Introduction of the liquid-liquid extraction

The year 1956 marked another milestone in the history of tantalum metal: despite some initial difficulties, H.C. Starck began its own production of tantalum oxide and potassium fluorotantalate (double fluoride) using a new technology, the so-called liquid-liquid extraction. In this process one dissolved substance is extracted from its solvent by another solvent, which is only slightly mixable with the first one.

Synthetic concentrates, ores and other materials were used as raw materials, which originally came from production in Goslar, but soon would be obtained from the electric furnace process in Laufenburg. Work with hydrofluoric acid turned out to be a special challenge in the liquid-liquid extraction because of related material problems. Luckily, solutions were found to these problems and only five years later, in 1961, the extraction was so successful that a decision was made to cease the production of tantalum oxide by the sodium hydroxide fusion method once and for all.

Prior to this, technological developments led to modifications in the Goslar plant. Two noteworthy changes were the extension of tantalum metal production in the marginal buildings V 95 to V 99 in 1955, and approval by the authorities in 1958 for production facilities for:

- synthetic tantalum and niobium concentrates
- potassium fluorotantalate
- tantalum and niobium metals
- tantalum and tantalum/niobium carbides and
- separating tantalum and niobium using liquid-liquid extraction deserves special mention.

During this time the old processes were gradually replaced by newer methods. For example, a new process called pot reduction for the production of tantalum metal was introduced in 1957. In this process double salt and sodium were arranged in alternating layers in a reaction boiler and subsequently ignited. Emptying the boiler afterwards caused a number of problems, which still evoke unpleasant memories today in those who were involved in the early days.

At the same time chemical reconcentration of furnace charges was abandoned in favor of the Laufenburg process (see description beginning on page 176), much to the relief of those affected at the plant because of the work involved.

The expansion of the tantalum business

With the dramatic expansion of capacity, for the first time ever, more than 100 tons of tantalum products were sold in 1965. This was largely due to the efforts of Hellwarth Lahusen, who, with the technical assistance of Dr. Herrmann Lang and, after Dr. Lang's death, Peter Borchers, developed and expanded the tantalum business. Since the early 1950s Lahusen had managed to establish and intensify contacts to European and American companies. These activities earned him the nickname "Mr. Tantalum" in international business circles.

The positive business development meant that the chemical facilities used in U 29/U 43 were "bursting at the seams" and therefore had to be moved to the "Halle West" production building. An application for the transfer of the tantalum and niobium production was submitted late in the summer of 1966. Approval was received at the beginning of 1968. This building had the advantage of having served as a warehouse "in reserve" since 1956, and thus the move went quickly.

In connection with the acquisition of the Ciba activities (for more information see the description of this acquisition beginning on page 179), another expansion soon followed. Among other things, this included the construction of a laboratory building (1969/1970) and the increase of sodium reduction capacities to four tons per month (1970).

These many changes first had to be integrated between 1970 and 1975. Technical expansion was taking place in several different production areas of the company during this time. In addition, the company was able to resolve bottlenecks in the extraction and oxide precipitation.

For many years H.C. Starck concentrated oxide and carbide production in "Halle West" building for compelling reasons: there was a downright "carbide boom" in those days. With sales of 260 tons of tantalum and niobium, the carbide business reached a temporary peak in 1976.

For reasons that will be explained later, one can only dream about such sales figures today. H.C. Starck's own potassium fluorotantalate production only served to cover the peak demand. The main quantities were covered by supplies from GfE.

The next technological advance

The year 1975 marked the beginning of a new era in the history of H.C. Starck's tantalum chemistry. As with the events described in the development of molybdenum and tungsten, there was increasing pressure to innovate because of the changed market situation. Customer demands in terms of quality increased dramatically over previous years. In particular, key customers in the capacitor industry, which was an important market for H.C. Starck, were soon no longer willing to accept standard grades.

One thing was certain: the installations in the old buildings on the outskirts of the plant could not be used for production of these higher grades. H.C. Starck thus decided to construct a new building to house the washing and formulation facilities for capacitorgrade tantalum metal powder. Reduction, on the other hand, still took place in the old building.

In addition, the first recycling facility began to take shape. The return of used and scrap capacitors steadily increased in line with the higher sales of powder for new capacitors. This justified the construction of a factory for the mechanized processing of tantalum capacitors.

At the same time, the requirements of analytical science increased dramatically in the wake of rapid developments in tantalum chemistry and especially in production processes for tantalum metal. Physical and chemical analyses were performed in two separate departments at H.C. Starck at the beginning of the 1970s. As a result, X-ray fluorescence and gas analyses were conducted by the physics department, while chemical analysis was taken care of in the laboratory for analytical chemistry. The staff of both the physics department and the analytical laboratory were constantly suffering from space problems.

Exorbitant metal prices rocked the market

Technology in the field of tantalum/niobium chemistry stagnated somewhat in the early 1980s. So the company concentrated on the elimination of weak spots, such as replacing outdated furnaces for oxide calcination by modern rotary tubular kilns. Investments were made also in environmental protection, including the expansion of the residual acid precipitation and the installation of a flue gas scrubber.

Struck by exorbitant metal prices, the branch fell into a so-called tantalum crisis in 1980 and 1981. Since many customers reacted uneasily, H.C. Starck was displeased with this development. In an effort to stabilize prices, the company temporarily supplied the market with tantalum raw materials.

Except for some slight fluctuations, the new "Halle West" worked to capacity during this time. Further expansion of capacity was not an issue because the Thai Tantalum Industrial Corporation (TTIC) project began to crystallize in 1979.

In 1977 H.C. Starck ceased buying its potassium fluorotantalate from GfE in favor of supplies from Mallinckrodt. This decision was directly related to H.C. Starck's acquisition of a 50-percent holding in NRC Inc. in Newton, Massachusetts.

NCR Inc., one of the most important producers of tantalum metal in the United States, bought its double salt from Mallinckrodt. Since the U.S. manufacturer could guarantee good supplies in terms of both quality and quantity, H.C. Starck's own production of potassium fluorotantalate in Goslar was temporarily shut down.

Scrap processing gains in importance

The late 1970s and early 1980s were characterized by increased recycling activities. A “Nissen hut” was set up at the plant to serve as a warehouse for tantalum scrap. Due to the increase in metal prices, recycling tantalum from used capacitors, cuttings, wire tailings and other scrap soon developed into an independent recycling operation.

In some cases, acids and acid mixtures had to be used to concentrate the scrap materials. The disposal of waste sulfuric acid, always left over after treating the plastic coverings of the used capacitors, caused some difficulties within the plant premises. The problem of disposing of waste sulfuric acid and other residual acids was ultimately solved with the introduction of an extensive waste water treatment system.

The production of tantalum metal

The so-called PL process for the production of tantalum metal was developed in Goslar in 1975. This process was based on the principle of kneading potassium fluorotantalate and sodium metal together with potassium chloride. However, it was soon discovered that the tantalum powder produced using this metal contained too much oxygen. In a subsequent deoxidization phase, this problem was more or less satisfactorily solved by adding calcium; later on (1979) magnesium was also used.

Other projects were less successful, for example, the attempt to convert the reduction to a continuous process, or the development of so-called B powders. Extensive research and development work, which was accelerated at H.C. Starck at the end of 1989, clearly showed that the last traces of alkalis in the ppm range could only be removed from capacitors produced with B powders with an unacceptable loss in capacitance. Thus the decision was made to switch to the process based on reduction in the salt melt, which was developed by the Japanese company V-Tech-Fansteel Inc. This move was linked to the acquisition of the V-Tech. The conversion to the V-Tech process was managed without problems. Additional input was provided by the Central Research and Development Division, which worked out details on doping, agglomeration and deoxidization.

In the mid-1980s H.C. Starck resumed production of potassium fluorotantalate, which had been discontinued several times in the course of the company's tantalum history. The crucial factor in making this decision was that the cost of purchasing potassium fluorotantalate had increased to such an extent that inhouse production now seemed more economically viable.

The decision was made to expand and modernize the “Halle West” building to accommodate double salt production. Waste water and flue gas management, which was thoroughly revised at the time, also benefited from this expansion.

After canceling the contract with Mallinckrodt, 100 percent of the potassium fluorotantalate requirements were covered by the company's own production. The Goslar factory also began to supply NRC, now H.C. Starck Inc., and later also H.C. Starck-V TECH. This arrangement still exists today.

No less than 50 percent of all the tantalum processed around the globe stems from H.C. Starck's plants in Goslar and Laufenburg, and the company's subsidiaries H.C. Starck Inc. in Newton, Massachusetts, in the United States and H.C. Starck-V TECH in Tokyo and Mito, Japan.

Niobium for special markets

While it is no exaggeration to say that H.C. Starck is the world's most important tantalum producer, the company only covers a relatively small market segment in the case of niobium. Worldwide consumption of its sister product is substantially higher than that of tantalum.

Niobium output currently amounts to some 20,000 tons of niobium oxide (Nb_2O_5) per year. The raw material stocks, especially the reserves in Brazil, are considered virtually inexhaustible. No less than 90 percent of the niobium requirements are allotted to the steel industry, which uses the metal in the form of ferroniobium in steel processing.

However, H.C. Starck has nothing to do with this market. The company's activities are concentrated on particularly promising specialized markets, "high-technology" industries in which high quality plays a crucial role. Included in this category are niobium oxides for electronic and optical applications, as well as niobium metal, alloys and carbides for superalloys and hardmetal or high-purity niobium chlorides for chemical vapor deposition (CVD).

After a business slump at the beginning of the 1990s, when sales of niobium specialties stagnated or even decreased, growth impulses from the electronics industry have since resulted in a distinct upward trend in business.

Among other things, the close connection to the application of passive electronic components – both through tantalum metal powder and niobium oxide – spurred product innovations in the 1990s, in which select niobates and tantalates with excellent dielectric properties were developed. Of these innovative products magnesium niobate, cobalt niobate and nickel niobate are currently establishing themselves in the market for multilayer ceramic capacitors and actuators.

Tantalum and niobium activities in Laufenburg

Failure to mention the Laufenburg activities as a part of the history of tantalum and niobium would be a serious omission. By 1954, in the early phase of the company's metal activities, the Rhina facility had already assumed a key role in raw material processing. The relatively inexpensive electricity then available in Laufenburg was once again a decisive factor.

Various raw materials which contain only a few percent of tantalum and niobium naturally, have been electrothermically processed to concentrates suitable for tantalum and niobium recovery since 1954. One must differentiate between two steps in this enrichment technology:

- 1) the production of an iron/tantalum/niobium-carbide alloy, especially from tin slags, but also from other raw materials, and
- 2) their possible further processing to oxidic concentrates, including the elimination of iron and other impurities to a large extent

From the beginning of August to the beginning of November 1954, the first fusion tests produced an alloy, which we sent on to Goslar for further concentration (see also page 166). The pilot plant work was blessed with success: larger batches of slags were melted between 1955 and 1956.

Only two years later, a group of employees joined together to form a development team for slag melting. The decisive factor behind this move was the purchase of 5,000 tons of Malaysian slags which had a tantalum content of 3.5 percent. Although the company had no practicable method of concentrating the slags at the time – because of their high titanium content these slags were considered more difficult to process than the slags used previously – H.C. Starck bought the apparently inexpensive shipment on the off-chance of success.

The stocks slags from Malaysia made quite an impressive “mountain” at the Laufenburg plant. They offered countless plants an excellent nutritive medium so that before long the stockpile was covered with vegetation. Many employees still recall the myriad of rare plants that adorned the slag stockpile. Tantalum was obviously not responsible for the exotic sight. It was the seeds brought in from tropical Malaysia that made this “botanical miracle” in Laufenburg possible.

The foreseeable problems with the high titanium content of the slags were soon evident in the course of the process development work, and the consequences were far less pleasant than nature’s botanical miracle. In addition to the metallic part on the bottom and the reduced slag on the top, a highly annoying intermediate layer formed during the melting process — much to the chagrin of the metallurgists. This layer had a major influence on results. Fortunately, the problem was soon eliminated with the help of the appropriate fluxes.

As a result of rapid technological developments, an expansion of production was soon necessary. Construction of a much larger production building (“Halle Süd II”) began in 1960. This building could accommodate two additional furnaces that were larger than the ones used previously. The first of these large-scale furnaces began production in November 1965.

The positive business development in the mid-1960s led to an euphoria that extended far beyond the factory gates. The company arranged for the rapid expansion of “Halle Süd” building. The startup of two additional large-capacity furnaces in 1967 was a further manifestation of this expansion course.

At peak times in those years, some 45 tons of slags were fed through the furnaces every day. This quantity corresponded to a monthly production of nearly 40 tons of tantalum. But H.C. Starck’s aim was not only to produce an adequate furnace charge, but also a further enriched oxidic concentrate that was comparable to natural ore.

Parallel to this work, the company performed reduction tests that culminated in 1961 in a pilot-plant production of syncon (an abbreviation for synthetic concentrates) in furnace S 1. Industrialscale production of these synthetic concentrates began in 1962, and as of 1966 furnace S 3 was used, partially at first, later entirely, for the production of syncon.

A business development with highs and lows

In the following years, synthetic concentrate increasingly established itself as the most important raw material for tantalum. It enabled H.C. Starck to produce starting materials for its chemical processes according to specific demand. In this way a certain independence could be gained from the producers’ tantalum ore mines and, as such, from raw material prices. Of course, the prerequisite was the “right” price for tin slags.

In 1960 the raw material input for slag melting was initially about 500 tons. This requirement increased dramatically so that by the mid-1960s quantities had climbed to between 10,000 and 12,000 tons per year. The world economic recession from 1971 to 1973 led to a strong downturn in these processing activities. A revival followed, and requests to concentrate slags for partners had a stabilizing effect.

The boom in the 1980s peaked in 1985 with a raw material input of nearly 17,000 tons. After a temporary lull, a new peak was reached in 1990, which was promptly followed by another downturn. Among other things, this slump was due to the fact that the available amount of tin slags containing tantalum decreased as a consequence of the dramatically falling tin prices and the subsequent production slowdown.

The acquisition of the rare metal facility

Favorable prognoses for the market development of certain metals in the 1950s and 1960s encouraged a number of companies to develop activities in sectors, which, knowing their product ranges today, no one would have expected. A good example is the former Ciba in Basle, Switzerland, which initiated a rare metal project in 1953 that concentrated on the metals tantalum and niobium. Since niobium, in particular, was predicted to have a bright future, a large department soon developed from the initially modestly equipped study group.

The pilot-plant operations in Basle, which began in a former sulfuric acid plant, soon focused on a chlorination facility for ores and a pilot distillation plant, as the experts had recognized that separation of tantalum/niobium chlorides through distillation was the best method. Tests with a number of different raw materials were performed in this chlorination plant.

Later, after they had mastered chlorination and separation of tantalum and niobium using distillation on a pilot plant scale, the process technology experts pursued both routes: oxides, which served as a preliminary stage in the production of carbides, and metal. In later years, up to 100 kilograms of tantalum and 20 kilograms of niobium per day were produced using fusion electrolysis.

When the expected market development for tantalum and niobium, in particular, failed to materialize in the mid-1960s, and Ciba was unwilling to use its know-how for expansion into the refractory metal sector, for example in tungsten, molybdenum or vanadium, the company decided to sell its activities.

Several months of negotiations with international companies did not lead to concrete results. Then H.C. Starck contacted Ciba in Basle, and subsequent talks were short and to the point.

The contract was signed on the second day of negotiations in June 1968. The plant and installations, patents, know-how and stocks were acquired for some 14 million Swiss francs. Hermann C. Starck himself did not see what he had actually purchased until a full month later.

The transfer to Goslar and Laufenburg

Two task forces were set up to prepare the integration of the plants and know-how in Goslar and Laufenburg. While Goslar was directly able to profit from Ciba's research and development, especially in the field of tantalum capacitor powders and carbide technology, Laufenburg benefited from the considerable expansion of its production facilities, including units for chlorination and distillation, an oxide facility and an electrolysis facility.

The necessary approval by the authorities was granted in only six months, by today's standards an amazingly short period of time. Thus the transfer of the plant equipment began by the turn of the year 1968/1969.

A laboratory facility that had been constructed at the ENAG plant shortly before was adjusted to the new requirements, which meant accommodating laboratories for analysis and development. However, the instruments of the analytical department were housed in the administration building of the ENAG plant. An existing warehouse was expanded to meet production needs.

With Ciba's permission production was initially continued in Basle. The transfer of plant and installations and the clearing of the buildings, including the new building constructed in 1963 and 1964, needed to be completed by mid-1969. This work had to be accomplished under extreme time pressure. Ciba's workshop staff and later, its production personnel, actively assisted in these ambitious efforts.

Despite all these problems, the following facilities and installations went on stream in Laufenburg in the course of 1969:

- the ore chlorination facility
- a pilot plant for chlorination of ferroalloys, which was soon supplemented by a second, improved plant
- distillation installations with a volumetric capacity of 2.5 to 3.0 tons
- oxide facilities with a total of four calcining furnaces, and
- electrolysis cells.
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Finding suitable staff turned out to be a major obstacle because there was an acute shortage of qualified personnel at the end of the 1960s. For almost 18 months the main burden rested on the Ciba production team, who started and maintained production in four shifts, while commuting back and forth between Basle and Laufenburg. It was 1971 before Ciba's personnel could gradually be pulled out.

While the startup of the chlorination and distillation facilities went satisfactorily, there were difficulties with the electrolysis. However, since there was a simultaneous strong drop in demand for coarse metal powder for fusion purposes, the electrolysis facilities were only operated temporarily and eventually shut down in 1975. Plans to reactivate the electrolysis in Goslar were also abandoned due to lack of demand. Thus the electrolytic production of tantalum metal ceased at H.C. Starck, as it did at other producers. In the meantime, the know-how for this process has largely been lost.

After the withdrawal of Ciba's personnel, H.C. Starck continued the expansion of the plant in the 1970s and 1980s with a small, but efficient team coordinated by highly capable production management. H.C. Starck also

- started up further distillation installations,
- shut down the oxide (ore) chlorination and concentrated instead on the chlorination of ferroalloys,
- improved distillation installations and expanded oxide units,
- transferred the pilot-plant and research facilities to the production building, and
- constantly improved the quality and purity of its tantalum and niobium oxides, not least by setting up cleanroom facilities.



The successes of the high and highest purity oxides ultimately helped promote internal competition. Thus the wet chemistry in Goslar sought to offer equally sophisticated products with its process. The result is a wide range of grades from which today's customers can benefit. In addition, chlorination technology was expanded for other metals in H.C. Starck's specialty program. Today, there are also facilities available for the production of various chlorides of tungsten and molybdenum.

Special attention has been paid to the environmentally relevant questions of, for example, flue gas and waste water. Right from the beginning, the production departments have endeavored to eliminate waste products of all kinds and to avoid their disposal in landfills. Slag fusion was a good method to accomplish this. The entire field of trace analyses in the context of the tantalum and niobium activities also gained a number of fundamentally new impulses during this time.

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